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(54) Title: IMPROVED VISUAL PROPERTIES FOR A WASH PROCESS

(57) Abstract: The present invention relates to compositions and methods for improving the visual properties of a wash process which utilizes lipophilic fluid (e.g., siloxane solvent) by coloring agents, sudsing agents and mixtures thereof.

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## **IMPROVED VISUAL PROPERTIES FOR A WASH PROCESS**

### **Related Applications**

This application claims priority under 35 U.S.C. § 119(e) to U.S. Provisional Application Serial Nos. 60/209,468, 60/209,250, 60/209,443 and 60/209,444 all of which were filed on June 5, 2000, and under 35 U.S.C. § 119(e) to U.S. Provisional Application Serial No. 60/247,530 filed on November 9, 2000.

### **Field of the Invention**

The present invention relates to compositions and methods for improving the visual properties of a wash process which utilizes lipophilic fluid (e.g., siloxane solvent). The present invention also relates to compositions and methods utilizing select colorants and/or sudsing agents (including suds producers and/or boosters) that provide improved visual aesthetics and/or cleaning signals to the user or operator of a lipophilic fluid (e.g., siloxane solvent) based wash process.

### **Background of the Invention**

For centuries, fabric articles have been washed using water-based processes. In the last century, this home chore was greatly simplified by the development of the automatic washing machine. However, while greatly simplifying the home laundry process, even the home laundry process using the automatic washing machine still requires a significant amount of presorting fabric articles by color and textiles. Typically whites are washed separately from colored fabrics, and brightly colored fabric articles (e.g., dark reds and blues) from less highly colored articles. Further sorting and handling is required when the fabric articles to be laundered include "dry clean only" articles.

More recently, water conservation efforts and environmental concerns have driven laundry machine manufacturers and laundry detergent manufacturers to reduce the amount of water required in the home laundry process. However, such efforts have focused on reducing water consumption by the wash medium rather than changing the wash medium from a primarily water based process.

In parallel, concerns have arisen around the use of "Perc" (short for perchloroethylene) as the wash medium for the commercial dry cleaning process. These concerns have lead to the development of a significant number of proposed alternatives the Perc-based processes, but to date all other alternatives are still not widely used. Examples include hydrocarbons and liquid carbon dioxide. A more recently proposed option as a replacement for Perc in the commercial dry cleaning field involves the use of siloxanes as the cleaning solution for "dry clean only" fabric articles (see, for example, US Patents: 5,942,007, August 24, 1999; 6,042,617 and 6,042,618, both March 28, 2000; 6,056,789, May 2, 2000; 6,059,845, May 9, 2000; and 6,063,135, May 16, 2000).

It has been discovered that further simplification of the automatic home laundry process and elimination of the reliance on a solely water based home laundry process are possible by using a lipophilic fluid based, for example siloxane based, wash medium for the home laundry process. This process allows not only the home cleaning of a consumer's "dry clean only" fabric articles, but also those "machine wash" articles conventionally washed at home in a water wash medium. Further while the consumer may still opt to wash such articles separately, the present invention process allows the consumer the freedom to significantly simplify the home laundry process by washing mixed loads of "dry clean only" and "machine wash" articles, thereby greatly reducing the presorting effort.

With such a dramatic change in the wash process comes the risk that the consumer's expectations, built up over generations of practicing the old water based system of laundering, will not be met -- not because of cleaning performance deficiencies but due simply to the fact that the process looks so different. Two visual cues to cleaning that consumers have come to expect are colorants in the products and sudsing during the wash. However, delivery of colorants to the lipophilic fluid systems useful for the new wash process, especially closed systems whereby the lipophilic fluid (e.g., siloxane

solvent) is recycled during the wash process and reused following a recovery process, creates a new world of problems and considerations for such a process. Careful consideration needs to be given to the potential for deposition of added colorants onto the fabrics, which under the old water wash system was not a concern (dye transfer from garment to garment in the water wash process was the major color deposition control issue).

Recycle of solvent further risks the appearance of trying to clean with "dirty" solution, from suspended soils being readily apparent in the wash medium. Considered addition of colorants to the lipophilic fluid to tinge or color the wash medium to mask an otherwise unsightly solution can further drive the acceptance of the system as an acceptable replacement for the old water based system.

Further, the lack of natural sudsing by a lipophilic fluid wash process (especially a siloxane wash process) and cleaning systems creates the need for select systems and/or specially added ingredients to permit the generation of suds to meet the expectations of the consumer. Careful consideration must also be given to avoiding negatively impacting the machine operations, or the ease of recycle or recovery of the lipophilic fluid, while designing the right sudsing profile to meet the consumer's needs.

#### Summary of the Invention

This need is met by the present invention wherein an automatic lipophilic fluid laundry process (e.g., a siloxane solvent laundry process) comprises the addition of one or more coloring agents, sudsing agents, or mixtures thereof.

The present invention also encompasses compositions useful in an automatic lipophilic fluid laundry process (e.g., a siloxane solvent laundry process) comprising one or more coloring agents, sudsing agents, or mixtures thereof.

The present invention further includes colored lipophilic fluid (e.g., siloxane solvent) systems useful in an automatic lipophilic fluid (e.g., siloxane solvent) laundry process, said solvent system comprising one or more lipophilic fluids (e.g., siloxane solvents) and one or more coloring agents safe for contact with fabrics during the automatic lipophilic fluid laundry process.

These and other aspects, features and advantages will become apparent to those of ordinary skill in the art from a reading of the following detailed description and the

appended claims. It is to be further understood that the present invention processes and compositions may be practiced outside of the home, including but not limited to institutional cleaning and commercial dry cleaning facilities. All percentages, ratios and proportions herein are by weight, unless otherwise specified. All temperatures are in degrees Celsius ( $^{\circ}$  C) unless otherwise specified. All measurements are in SI units unless otherwise specified. All documents cited are in relevant part, incorporated herein by reference.

#### Detailed Description of the Invention

The present invention encompasses an automatic lipophilic fluid laundry process, including automatic siloxane solvent laundry processes, typically a home laundry process, comprising the step of cleaning fabric articles in an automatic washing machine (typically in the home) with a wash medium comprising lipophilic fluid (e.g., siloxane) and one or more materials selected from coloring agents, sudsing agents, and mixtures thereof.

The present invention also relates to an automatic laundry process (typically in the home) comprising contacting, in an automatic washing machine, machine washable fabric articles with a wash medium comprising lipophilic fluid (e.g., siloxane) and one or more materials selected from coloring agents, sudsing agents, and mixtures thereof. More preferred is the lipophilic fluid-containing (e.g., siloxane-containing) wash medium comprising less than about 50% water, but in some preferred executions it is desired to include some amount of purposively added water preferably in the range of from about 0.1% to about 50% water by weight of the wash medium. In yet another embodiment the system is essentially free of water.

Further preferred processes of the present invention are directed to an automatic laundry (typically in the home) process comprising contacting, in an automatic washing machine, a mixed load of fabric articles comprising machine washable and dry clean only fabric articles with a wash medium comprising lipophilic fluid (e.g., siloxane) and one or more materials selected from coloring agents, sudsing agents, and mixtures thereof.

The terms "fabrics" and "fabric articles", as used herein, are intended to mean any article that is customarily cleaned in a conventional laundry process or in a dry cleaning process. As such the term encompasses articles of clothing, linen, drapery, and clothing

accessories. The term also encompasses other items made in whole or in part of fabric, such as tote bags, furniture covers, tarpaulins and the like.

The term “machine washable fabric articles”, as used herein, means those fabric articles readily identified by the fabric industry and consumers as safe for laundering by a conventional aqueous automatic home laundry process. Consumers are frequently helped in this identification of fabric articles by manufacturer’s tags indentifying the fabric article as “machine washable” or some similar description.

The term “dry clean only fabric articles”, as used herein, means those fabric articles readily identified by the fabric industry and consumers as unsafe for laundering by a conventional aqueous automatic home laundry process, and instead requiring special handling with a conventional non-aqueous solvent such as Perc. Again, consumers are frequently helped in this identification of fabric articles by manufacturer’s tags indentifying the fabric article as “dry clean only” or some similar description.

The phrase “dry weight of a fabric article” as used herein means the weight of a fabric article that has no intentionally added fluid weight.

The phrase “absorption capacity of a fabric article” as used herein means the maximum quantity of fluid that can be taken in and retained by a fabric article in its pores and interstices. Absorption capacity of a fabric article is measured in accordance with the following Test Protocol for Measuring Absorption Capacity of a Fabric Article.

**Test Protocol for Measuring the Absorption Capacity of a Fabric Article**

Step 1: Rinse and dry a reservoir or other container into which a lipophilic fluid will be added. The reservoir is cleaned to free it from all extraneous matter, particularly soaps, detergents and wetting agents.

Step 2: Weigh a “dry” fabric article to be tested to obtain the “dry” fabric article’s weight.

Step 3: Pour 2L of a lipophilic fluid at ~20C into the reservoir.

Step 4: Place fabric article from Step 2 into the lipophilic fluid-containing reservoir.

Step 5: Agitate the fabric article within the reservoir to ensure no air pockets are left inside the fabric article and it is thoroughly wetted with the lipophilic fluid.

Step 6: Remove the fabric article from the lipophilic fluid-containing reservoir.

Step 7: Unfold the fabric article, if necessary, so that there is no contact between same or opposite fabric article surfaces.

Step 8: Let the fabric article from Step 7 drip until the drop frequency does not exceed 1 drop/sec.

Step 9: Weigh the “wet” fabric article from Step 8 to obtain the “wet” fabric article’s weight.

Step 10: Calculate the amount of lipophilic fluid absorbed for the fabric article using the equation below:

$$FA = (W-D)/D*100$$

where:

FA = fluid absorbed, % (i.e., the absorption capacity of the fabric article in terms of % by dry weight of the fabric article)

W = wet specimen weight, g

D = initial specimen weight, g

By the term "non-immersive" it is meant that essentially all of the fluid is in intimate contact with the fabric articles. There is at most minimal amounts of "free" wash liquor. It is unlike an "immersive" process where the washing fluid is a bath in which the fabric articles are either submerged, as in a conventional vertical axis washing machine, or plunged into, as in a conventional horizontal washing machine. The term "non-immersive" is defined in greater detail according to the following Test Protocol for Non-Immersive Processes. A process in which a fabric article is contacted by a fluid is a non-immersive process when the following Test Protocol is satisfied.

#### Test Protocol for Non-Immersive Processes

Step 1: Determine absorption capacity of a fabric specimen using Test Protocol for Measuring Absorption Capacity of a Fabric Article, described above.

Step 2: Subject a fabric article to a fluid contacting process such that a quantity of the fluid contacts the fabric article.

Step 3: Place a dry fabric specimen from Step 1 in proximity to the fabric article of Step 2 and move/agitate/tumble the fabric article and fabric specimen such that fluid transfer from the fabric article to the fabric specimen takes place (the fabric article and fabric specimen must achieve the same saturation level).

Step 4: Weigh the fabric specimen from Step 3.

Step 5: Calculate the fluid absorbed by the fabric specimen using the following equation:

$$FA = (W-D)/D*100$$

where:

FA = fluid absorbed, %

W = wet specimen weight, g

D = initial specimen weight, g

Step 6: Compare the fluid absorbed by the fabric specimen with the absorption capacity of the fabric specimen. The process is non-immersive if the fluid absorbed by the fabric specimen is less than about 0.8 of the absorption capacity of the fabric specimen.

The term "automatic home laundry process", as used herein, means the laundry process as practiced by the consumer using an automatic washing machine, preferably located within the consumer's residence (herein referred to as the process being conducted "in the home"), but also including public laundromats whereby the consumer follows essentially the same laundry process as though the automatic washing machine were present in the home.

The term "automatic lipophilic fluid laundry process", as used herein, means the laundry process as practiced using an automatic washing machine, preferably located in the home, but also including public Laundromats, institutional and commercial laundry facilities, and whereby the laundry process comprises (as some or all of the process) the contacting of the fabrics to be treated in the process with a lipophilic fluid-containing wash medium. Likewise, the term "automatic siloxane solvent laundry process", as used herein, means the laundry process as practiced using an automatic washing machine, preferably located in the home, but also including public Laundromats, institutional and commercial laundry facilities, and whereby the laundry process comprises (as some or all of the process) the contacting of the fabrics to be treated in the process with a siloxane solvent-containing wash medium.

The term "wash medium", as used herein, means the liquid [including but not limited to, fluid(s) and/or solution(s) and/or solvent(s) and/or emulsion(s)] which is used to wet the fabric articles in the wash load during the automatic laundry process.

The term "lipophilic solvent" as used herein means any non-aqueous fluid capable of removing sebum, as qualified by the test described herein after.

The terms "siloxane" or "siloxane solvents", as used herein, means lipophilic fluids which are silicone fluids that are non-polar and insoluble in water. They are completely miscible in typical aliphatic and aromatic solvents, including the halogenated solvents, but are only partially miscible with the intermediate petroleum fractions such as naphthalenes. Linear siloxanes (see for example US Patents 5,443,747, and 5,977,040, both incorporated herein by reference in their entirety) and cyclic siloxanes are useful herein, including the cyclic siloxanes selected from the group consisting of octamethylcyclotetrasiloxane (tetramer), dodecamethyl-cyclohexasiloxane (hexamer), and preferably decamethyl-cyclopentasiloxane (pentamer, commonly referred to as "D5"). Most



preferred are wash mediums wherein the siloxane comprises more than about 50% cyclic siloxane pentamer, more preferably more than about 75%, most preferably at least about 90% of the pentamer. Also preferred are wash mediums comprising siloxanes which are a mixture of cyclic siloxanes having at least about 90% (preferably at least about 95%) pentamer and less than about 10% (preferably less than about 5%) tetramer and/or hexamer, with less than about 1% of tetramer being most preferred.

For the present invention process, the specific method for contacting the wash medium containing the lipophilic fluid (e.g., siloxane) with the fabric article may be any method which results in complete wetting of the fabric articles in the wash load by the automatic washing machine, in contrast to spot wetting and/or hand wetting of the fabric articles. This includes contacting the fabric articles in an immersive bath of the wash medium or, preferably, using lower volumes of wash medium as is possible by low volume wetting means such as spraying to uniformly wet the fabric articles. In one embodiment the fabric articles are wetted with the wash medium only to the extent of less than about 500%, or less than about 300%, by weight of the fabric articles in the wash load. Another embodiment includes contacting the fabric articles using a method that comprises at least one non-immersive step whereby the fabric article is wetted with the wash medium (e.g., by uniform spraying) for example only to the extent of less than about 200% (or less than about 150%) by weight of the fabric article in the wash load.

One aspect of the present invention involves the contacting of the fabric articles with a wash medium comprising less than about 50% water, more preferably less than about 30%, less than about 20%, less than about 10%, less than about 5%, less than about 1%, and most preferably no purposively added water. However, in another aspect of the present invention some amount of purposively added water is a part of the wash medium with the lipophilic fluid (e.g., siloxane), including for example either immiscible with the lipophilic fluid or as an emulsion comprising lipophilic fluid (e.g., siloxane), water, and typically an emulsifying agent. Preferably the water comprises from about 0.1% to about 50%, more preferably from about 0.1% to about 30%, from about 0.1% to about 20%, from about 0.1% to about 10%, from about 0.1% to about 5%, and from about 0.1% to about 1%.

Obviously the present invention process cannot use current conventional automatic washing machines as developed for aqueous wash processes. While automatic washing machines useful for the present process may be connected to a water source, such connection for purposes of carrying out the current process is typically for the supply of the optional purposively added water. A supply of the lipophilic fluid (e.g., siloxane) is necessary, preferably stored in a container for use in the current process and into which the lipophilic fluid is returned (following a suitable cleaning process) after contact with the fabric articles in the automatic laundry process, preferably an automatic home laundry process.

While an apparatus having the various components as defined for the immersive commercial dry cleaning process as described in US Patent 6,059,845, issued May 9, 2000, and US Patent 6,063,135, issued May 16, 2000 (both incorporated herein by reference in their entirety), if modified for residential size and consumer maintenance, could be used to practice the present invention process, an immersive process for the present invention is not preferred. Reasons include the constraints (versus the commercial dry cleaner size and operation taught in these patents) associated with supplying, storing and cleaning larger volumes of siloxane in the home.

For these reasons lower volume processes such as those utilizing a uniform spray process which completely wets the fabric articles with the lower volumes of wash medium as described herein before are highly preferred. For example modifications of conventional low water wash appliances to deliver low levels of siloxane-containing wash medium rather than a water wash medium should be considered; such conventional water wash appliances are described for example in US Patents: 4,489,574; 4,489,455; 5,191,669; 5,191,668; 5,233,718; and 5,671,494, all incorporated herein by reference in their entirety. A most preferred automatic washing machine useful for this low volume process is described in detail in the co-filed, copending patent application, P&G Case 8119P, U.S. Serial Number 60/209,468, filed June 5, 2000, incorporated herein by reference in its entirety. Further preferred are automatic washing machines which guarantee homogeneous coverage of the fabric articles with the lipophilic fluid (e.g., siloxane) -containing wash medium by intermittent spin and spray followed by random tumbling until all the wash medium has been sprayed. Also preferred are such machines

which reuse the siloxane-containing wash medium via an immediate re-use/recycling action, for example by passing the wash medium over a particle removal filter after extraction from the fabric articles and then immediately spraying it back onto the fabric articles.

Further preferred automatic washing machines for practicing the present invention processes are designed to also dry the fabric articles in the same apparatus. This not only allows the consumer the convenience of not having to handle the wet fabric articles but also permits recovery of all the siloxane for reuse or cleaning.

It is also envisioned that adjuncts for cleaning and/or treating the fabric articles according to the desires of the consumer may be added to the process. The automatic washing machine therefore preferably includes recepticals for receiving and/or dispensing such adjuncts into the automatic laundry process at the desired time, either with the wash medium or separate from it. Cartridges containing such adjuncts (either through refilling or purchased with the adjunct) which releaseably attach to the machine are also optional executions. A particularly preferred adjunct is any material which functions as an antistatic agent when combined with the lipophilic fluid-containing (e.g., siloxane-containing) wash medium in the present automatic laundry process.

An additional preferred feature of the automatic washing machine is the ability to clean and reuse the lipophilic fluid (e.g., siloxane) for more than one automatic laundry process. A preferred means for cleaning the lipophilic fluid for multiple uses is a replaceable filter. Such filter should preferably include filter materials capable of removing and collecting at least the body soils removed from the fabric articles during the automatic home laundry process. Activated charcoal, silicas, molecular sieves, and/or hydrophobically modified papers are just some optional components of such filters. The attachment to the automatic washing machine is preferably by means such that the consumer can readily replace it at regular intervals.

#### Lipophilic Fluid

The lipophilic fluid herein is one having a liquid phase present under operating conditions. In general such a fluid can be fully liquid at ambient temperature and pressure, can be an easily melted solid, e.g., one which becomes liquid at temperatures in the range from about 0 deg. C to about 60 deg. C, or can comprise a mixture of liquid and

vapor phases at ambient temperatures and pressures, e.g., at 25 deg. C and 1 atm. pressure. Thus, the essential lipophilic fluid is not a compressible gas such as carbon dioxide. It is preferred that the lipophilic fluid herein be inflammable or, have relatively high flash points and/or low VOC characteristics, these terms having their conventional meanings as used in the dry cleaning industry, to equal or, preferably, exceed the characteristics of known conventional dry cleaning fluids.

Suitable lipophilic fluids herein readily flow and are non-viscous. In general, the lipophilic fluids herein are required to be fluids capable of at least partially dissolving sebum or body soil as defined in the test hereinafter. Mixtures of lipophilic fluid are also suitable, and provided that the requirements of the test are met, the lipophilic fluid can include any fraction of dry-cleaning solvents, especially newer types including non-fluorinated solvents, or perfluorinated amines. Some perfluorinated amines such as perfluorotributylamines while unsuitable for use as lipophilic fluid may be present as one of many possible adjuncts present in the lipophilic fluid. Other suitable lipophilic fluids include diol solvent systems e.g., higher diols such as C6- or C8- or higher diols; organosilicon solvents including both cyclic and acyclic types, and the like; and mixtures thereof.

A preferred group of nonaqueous liquids suitable for incorporation as the major component of the lipophilic fluid includes low-volatility non-fluorinated organics, silicones, especially those other than amino-functional silicones, and mixtures thereof. Low volatility nonfluorinated organics include for example OLEAN and other polyol esters, or certain relatively nonvolatile biodegradable mid-chain branched petroleum fractions. Suitable silicones for use as a major component, e.g., more than 50%, of the lipophilic fluid include cyclopentasiloxane, sometimes termed "D5", or linear analogs having approximately similar volatility, optionally complemented by other compatible silicones. Suitable silicones are well known in the literature, see, for example, Kirk Othmer's Encyclopedia of Chemical Technology, and are available from a number of commercial sources, including General Electric, Toshiba Silicone, Bayer, and Dow Corning. Other suitable fluids are commercially available from Procter & Gamble or from Dow Chemical and other suppliers. For example one suitable silicone is SF-1528

available from GE silicone fluids. It is worth noting that SF-1528 fluid is 90% cyclopentasiloxane (D5).

Depending on the nature of treatment the lipophilic fluid may be removed mechanically, evaporatively, or any combination thereof. For example, if the purpose of the treatment is to provide cleaning it will be desirable to mechanically remove from the fabric articles at least 50% of the textile treatment liquid, for example by spinning. On the other hand, if the purpose of the treatment is to deposit a conditioning agent into the fabric, the liquid is preferably removed evaporatively.

#### Qualification of Lipophilic Fluid -- Lipophilic Fluid Test (LF Test)

Any non-aqueous fluid that is both capable of meeting known requirements for a dry-cleaning fluid (e.g, flash point etc.) and is capable of at least partially dissolving sebum, as indicated by the test method described below, is suitable as a lipophilic fluid herein. The ability of a particular material to remove sebum can be measured by any known technique. As a general guideline, perfluorobutylamine (Fluorinert FC-43®) on its own (with or without adjuncts) is a reference material that, by definition, is unsuitable as the lipophilic fluid herein (it is essentially a non-solvent) while D5 dissolves sebum.

The following is the method for investigating and qualifying other materials, e.g., other low-viscosity, free-flowing silicones, for use as the lipophilic fluid. The method uses commercially available Crisco ® canola oil, oleic acid (95% pure, available from Sigma Aldrich Co.) and squalene (99% pure, available from J.T. Baker) as model soils for sebum. The test materials should be substantially anhydrous and free from any added adjuncts, or other materials during evaluation.

Prepare three vials. Place 1.0 g of canola oil in the first; in a second vial place 1.0 g of the oleic acid (95%), and in a third and final vial place 1.0g of the squalene (99.9%). To each vial add 1 g of the fluid to be tested for lipophilicity. Separately mix at room temperature and pressure each vial containing the lipophilic soil and the fluid to be tested for 20 seconds on a standard vortex mixer at maximum setting. Place vials on the bench and allow settling for 15 minutes at room temperature and pressure. If, upon standing, a single phase is formed in any of the vials containing lipophilic soils, then the fluid qualifies as suitable for use as a "lipophilic fluid" in accordance with the invention.

However, if two or more separate layers are formed in all three vials, then the amount of fluid dissolved in the oil phase will need to be further determined before rejecting or accepting the fluid as qualified.

In such a case, with a syringe, carefully extract a 200 microliter sample from each layer in each vial. The syringe-extracted layer samples are placed in GC autosampler vials and subjected to conventional GC analysis after determining the retention time of calibration samples of each of the three models soils and the fluid being tested. If more than 1% of the test fluid by GC, preferably greater, is found to be present in any one of the layers which consists of the oleic acid, canola oil or squalene layer, then the test fluid is also qualified for use as a lipophilic fluid. If needed, the method can be further calibrated using heptacosafuorotributylamine, i.e., Fluorinert FC-43 (fail) and cyclopentasiloxane (pass).

A suitable GC is a Hewlett Packard Gas Chromatograph HP5890 Series II equipped with a split/splitless injector and FID. A suitable column used in determining the amount of lipophilic fluid present is a J&W Scientific capillary column DB-1HT, 30 meter, 0.25mm id, 0.1um film thickness cat# 1221131. The GC is suitably operated under the following conditions:

Carrier Gas: Hydrogen

Column Head Pressure: 9 psi

Flows: Column Flow @ ~1.5 ml/min.

Split Vent @ ~250-500 ml/min.

Septum Purge @ 1 ml/min.

Injection: HP 7673 Autosampler, 10 ul syringe, 1ul injection

Injector Temperature: 350 °C

Detector Temperature: 380 °C

Oven Temperature Program: initial 60 °C, hold 1 min.

rate 25 °C/min.

final 380 °C hold 30 min.

Preferred lipophilic fluids suitable for use herein can further be qualified for use on the basis of having an excellent garment care profile. Garment care profile testing is

well known in the art and involves testing a fluid to be qualified using a wide range of garment or fabric article components, including fabrics, threads and elastics used in seams, etc., and a range of buttons. Preferred lipophilic fluids for use herein have an excellent garment care profile, for example they have a good shrinkage or fabric puckering profile and do not appreciably damage plastic buttons.

For purposes of garment care testing or other qualification, e.g., flammability, a lipophilic fluid for use in the lipophilic fluid can be present in a mixture, e.g., with water, at approximately the ratio to be used in the final lipophilic fluid which will come into contact with fabric articles. Certain materials, which remove sebum, qualify for use as lipophilic fluids; for example, ethyl lactates can be quite objectionable in their tendency to dissolve buttons, and if such a material is to be used in the lipophilic fluid, it will be formulated with water and/or other solvents such that the overall mix is not substantially damaging to buttons. Other lipophilic fluids, D5 for example, meets the garment care requirements commendably. Some suitable lipophilic fluids may be found in granted U.S. Patent Nos. 5,865,852; 5,942,007; 6,042,617; 6,042,618; 6,056,789; 6,059,845; and 6,063,135, which are herein incorporated by reference.

Lipophilic solvents can include linear and cyclic polysiloxanes, hydrocarbons and chlorinated hydrocarbons. More preferred are the linear and cyclic polysiloxanes and hydrocarbons of the glycol ether, acetate ester, lactate ester families. Preferred lipophilic solvents include cyclic siloxanes having a boiling point at 760 mm Hg. of below about 250°C. Specifically preferred cyclic siloxanes for use in this invention are octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane. It should be understood that useful cyclic siloxane mixtures might contain, in addition to the preferred cyclic siloxanes, minor amounts of other cyclic siloxanes including hexamethylcyclotrisiloxane or higher cyclics such as tetradecamethylcycloheptasiloxane. Generally the amount of these other cyclic siloxanes in useful cyclic siloxane mixtures will be less than about 10 percent based on the total weight of the mixture.

#### Coloring Agents:

"Coloring agents", as used herein, are any substance (including dyes and pigments) added solely for the purpose of imparting color to a composition to be used in

an automatic lipophilic fluid (e.g., siloxane) laundry process, including the lipophilic fluid (e.g., siloxane solvent) wash medium composition, while being safe for contact with all fabrics in contact with the lipophilic fluid wash medium during the automatic lipophilic fluid laundry process. The safety of coloring agents for the present invention compositions and processes can be determined by contacting white fabric with potential coloring agents in an automatic lipophilic fluid laundry process, whereby coloring agents safe for use therein result in very little or no observable coloring of the white fabrics, as determined by the following test:

- white fabric swatches of various fabric content (3 of each) are used as tracers for dye transfer from the coloring agents;
- each fabric swatch is measured to determine a baseline value on the Hunter Whiteness Scale;
- measurements are performed using a spectrophotometer of choice that generates an illuminant of C/2° ;
- values are recorded as L<sub>h</sub>, a<sub>h</sub>, and b<sub>h</sub>;
- the swatches are subsequently washed and dried in the presence of the potential coloring agent(s);
- spectrophotometric measurements of the swatch tracers are taken at the end of predetermined cycles;
- differences are calculated between the initial and final cycle and recorded as delta E values;
- $\text{delta E} = \text{square root } \{(L_{h_i} - L_{h_f})^2 + (a_{h_i} - a_{h_f})^2 + (b_{h_i} - b_{h_f})^2\}$ ;
- a noticeable difference for 3 or more cycles is defined as a delta E of  $\geq 5$ .

It is to be recognized that coloring agents do not include those active ingredients added for purposes other than to impart color to such compositions or wash processes. For example, surfactants typically are slightly colored due to some low level of impurities; photobleaches typically give solutions a blue tint; brighteners can also provide some coloring. Thus, while the present invention compositions and processes typically include such active ingredients, the coloring agents according to the present invention are added with them, typically to mask or complement any color produced by such active ingredients.



While coloring agents generally are well known, their selection for use in the present invention processes and compositions, especially siloxane solvent systems, require careful consideration of the wash process conditions and wide range of typical fabrics which may be in contact with the coloring agent, as well as the purpose for which the coloring agent is being used. In particular, coloring agents to be used in a composition to be added to the lipophilic fluid, (e.g., siloxane solvent) during the wash process typically will be present in the composition to provide the desired aesthetics to the compositions. Such compositions typically will be added to deliver cleaning agents (e.g., surfactants, polymers, enzymes, bleaching agents) and/or fabric treatment agents (e.g., softening agents, antistatic agents, brighteners) to the wash process. The coloring agents thus provide a more consumer desirable composition, such as a blue liquid laundry detergent or a green speckled laundry powder.

Such coloring agents not only must be stable in such compositions and compatible with the other adjunct ingredients the coloring agents comes in contact with, but also must not deposit from the wash medium onto the fabrics in contact with the wash medium (i.e., they must be safe as defined by the test provided herein before). It is also highly desirable that the coloring agents used in such compositions are removable (completely or at least in major part) by the lipophilic fluid (especially a siloxane solvent) recovery system of the automatic laundry machine being used for the process (e.g., by filtration and/or distillation), and/or that the coloring agents are compatible with any coloring agent system used to color the lipophilic fluid system being used in the process, and/or the coloring agents are readily destroyed (completely or at least in major part) by other conditions utilized during the wash process. The destruction of the coloring agents may be by any means, including but not limited to chemical (e.g., oxidative and/or reductive reactions), mechanical (e.g., ultrasound), electrical, and/or thermal degradation. This includes, for example, destroying coloring agents by the use of ozone in the system, or during a heating step such as drying of the fabrics. A further consideration when selecting the coloring agent is the color compatibility with other adjunct active ingredients that provide some color.

Use of coloring agents as a colorant for the lipophilic fluid (e.g., siloxane) wash solution have similar requirements as above for the coloring agents used in the

compositions added into the system – they must be at least partially stable in the wash process (to the extent necessary to provide the desired coloring of the wash medium during at least a portion of the wash process) and also must not deposit from the wash medium onto the fabrics in contact with the wash medium (i.e., they must be safe as defined by the test provided herein before). It is desirable for such coloring agents to be stable to the recycle and recovery processes employed by the automatic machine, but this is not necessary if the composition added to the wash medium with each load of laundry includes coloring agents at a level sufficient to provide the desired tinting of the wash medium or by some other replenishment process.

It is also possible to design coloring systems whereby the coloring agents used to tint the lipophilic fluid (e.g., siloxane solvent) and the coloring agents formulated into the composition added with each laundry load work cooperatively. For example, the coloring agents from the added compositions may be formulated to match the coloring agents used to tint the lipophilic fluid (e.g., siloxane solvent) system, which would be desirable to replenish lipophilic fluid-tinting colorants lost in part by the previous wash cycle(s). It is also possible to design a color system whereby the coloring agents added with the composition change the color of the lipophilic fluid (e.g., siloxane) wash medium, but are subsequently removed at some stage in the wash process (e.g., the siloxane tint undergoes a color change when the ozone treatment step is reached; the solvent changes back to the original color when the solvent recovery process is complete).

Further, it is possible to utilize coloring systems whereby the coloring agents are selectively soluble in the lipophilic fluid and/or selectively soluble in water, which may be desirable when using an aqueous emulsion (e.g., a siloxane emulsion) as the wash medium. This may be useful for easily monitoring the state of the emulsion, including the ability to observe the water and lipophilic fluid phases when the emulsion is broken.

#### Sudsing Agents:

The lack of natural sudsing by the lipophilic fluids (especially siloxane) wash process and cleaning systems creates the need for select systems and/or specially added ingredients to permit the generation of suds to meet the expectations of the consumer. As used herein, “sudsing agents” means any compounds and/or mixtures of compounds which when included in the lipophilic fluid (e.g., siloxane) wash medium generate

observable sudsing effect during the operation of the automatic laundry machine.

Sudsing agents therefore also include materials which act as suds or foam boosters, as well as select surfactants and/or emulsifiers which have sudsing properties. Properties of foams generally are very well known, having been described for example in "Additives for Foams", by Lai and Dixit, at page 315 et. Seq. of Foams, Theory, Measurements, and Applications (copyright 1996 by Marcel Dekker, Inc., N.Y.; Prud'homme and Khan, editors).

In particular, sudsing agents may be introduced into the lipophilic fluid wash process either by the addition of a composition containing one or more sudsing agents, such compositions typically are added to deliver cleaning agents (e.g., surfactants, polymers, enzymes, bleaching agents) and/or fabric treatment agents (e.g., softening agents, antistatic agents, brighteners) to the wash process, or by the addition of a separate sudsing agent composition (similar to the separate addition of a "rinse aide" in an automatic diswashing machine). Such sudsing agents not only must be stable in such compositions and compatible with the other adjunct ingredients the sudsing agent comes in contact with, but also must not negatively impact the fabric if it deposits from the wash medium onto the fabrics in contact with the wash medium.

It is also highly desirable that the sudsing agents are removable (completely or at least in major part) by the lipophilic fluid (e.g., siloxane solvent) recovery system of the automatic laundry machine being used for the process (e.g., by filtration and/or distillation), and/or are readily destroyed (completely or at least in major part) by other conditions utilized during the wash process. The destruction of the sudsing agents may be by any means, including but not limited to chemical (e.g., oxidative and/or reductive reactions), mechanical (e.g., ultrasound), electrical, and/or thermal degradation. This includes, for example, destroying sudsing agents by the use of ozone in the system, or during a heating step such as drying of the fabrics. Alternatively, if the sudsing agent is to be reusable after recovery of the lipophilic fluid, it is desirable for such sudsing agents to be stable to the recycle and recovery processes employed by the automatic machine. Complete stability is not necessary if the composition added to the wash medium with each load of laundry includes suding agents at a level sufficient to replenish the quantity of sudsing agent lost by previous wash cycles.

Careful consideration must also be given to avoiding negatively impacting the machine operations, or the ease of recycle or recovery of the lipophilic fluid (e.g., siloxane solvent), while designing the right sudsing profile to meet the consumer's needs. It is therefore necessary to consider the type of machine and process being utilized to determine the level of sudsing agent in the wash medium. For example, a process that utilizes spray jets for the dispersion of the wash medium onto the fabric is likely to generate more foaming from lower concentrations of sudsing agents than an immersive bath process that utilizes only a rotating drum for agitation of the wash medium. One of ordinary skill in the art can readily select the levels needed to match such sudsing properties to the machine and process being used.

Finally, compositions containing sudsing agents may comprise added water, either immiscible or miscible (e.g., as an emulsion) with the lipophilic fluid. Typically the water is added to the lipophilic fluid-containing wash medium (e.g., siloxane wash medium) at a level of at least about 0.1% (or at least about 0.5%, or at least about 1%) with the sudsing agents.

#### Adjunct Ingredients

Adjunct materials can vary widely and can be used at widely ranging levels. For example, deterative enzymes such as proteases, amylases, cellulases, lipases and the like as well as bleach catalysts including the macrocyclic types having manganese or similar transition metals all useful in laundry and cleaning products can be used herein at very low, or less commonly, higher levels. Adjunct materials that are catalytic, for example enzymes, can be used in "forward" or "reverse" modes, a discovery independently useful from the specific appliances of the present invention. For example, a lipolase or other hydrolase may be used, optionally in the presence of alcohols as adjuncts, to convert fatty acids to esters, thereby increasing their solubility in the lipophilic fluid. This is a "reverse" operation, in contrast with the normal use of this hydrolase in water to convert a less water-soluble fatty ester to a more water-soluble material. In any event, any adjunct ingredient must be suitable for use in combination with the lipophilic fluid.

Some suitable cleaning additives include, but are not limited to, builders, surfactants, enzymes, bleach activators, bleach catalysts, bleach boosters, bleaches,

alkalinity sources, antibacterial agents, perfumes, pro-perfumes, finishing aids, lime soap dispersants, composition malodor control agents, odor neutralizers, polymeric dye transfer inhibiting agents, crystal growth inhibitors, photobleaches, heavy metal ion sequestrants, anti-tarnishing agents, anti-microbial agents, anti-oxidants, anti-redeposition agents, soil release polymers, electrolytes, pH modifiers, thickeners, abrasives, divalent or trivalent ions, metal ion salts, enzyme stabilizers, corrosion inhibitors, diamines or polyamines and/or their alkoxylates, solvents, process aids, fabric softening agents, optical brighteners, hydrotropes, fabric softeners, antistatic agents, dye fixatives, dye abrasion inhibitors, anti-crooking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, soil repellency agents, sunscreen agents, anti-fade agents, and mixtures thereof.

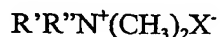
Emulsifiers are particularly desired adjuncts when the wash medium is an aqueous siloxane emulsion, and are well known in the chemical art. Essentially, an emulsifier acts to bring two or more insoluble or semi-soluble phases together to create a stable or semi-stable emulsion. It is preferred in the claimed invention that the emulsifier serves a dual purpose wherein it is capable of acting not only as an emulsifier but also as a treatment performance booster. For example, the emulsifier may also act as a surfactant thereby boosting cleaning performance. Both ordinary emulsifiers and emulsifier/surfactants are commercially available.

The term "surfactant" conventionally refers to materials that are surface-active either in the water, the lipophilic fluid, or the mixture of the two. Some illustrative surfactants include nonionic, cationic and silicone surfactants as used in conventional aqueous detergent systems. Suitable nonionic surfactants include, but are not limited to:

- a) Polyethylene oxide condensates of nonyl phenol and myristyl alcohol, such as in US 4685930 Kasprzak; and
- b) fatty alcohol ethoxylates,  $R-(OCH_2CH_2)_aOH$   $a=1$  to 100, typically 12-40,  $R=$  hydrocarbon residue 8 to 20 C atoms, typically linear alkyl. Examples polyoxyethylene lauryl ether, with 4 or 23 oxyethylene groups; polyoxyethylene cetyl ether with 2, 10 or 20 oxyethylene groups; polyoxyethylene stearyl ether, with 2, 10, 20, 21 or 100 oxyethylene groups; polyoxyethylene (2), (10) oleyl ether, with 2 or 10 oxyethylene groups.

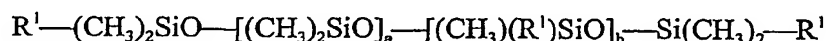
Commercially available examples include, but are not limited to: ALFONIC, BRIJ, GENAPOL, NEODOL, SURFONIC, TRYCOL. See also US 6013683 Hill et al.,.

Suitable cationic surfactants include, but are not limited to dialkyldimethylammonium salts having the formula:

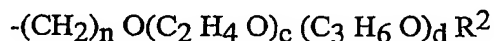


Where each R'R'' is independently selected from the group consisting of 12-30 C atoms or derived from tallow, coconut oil or soy, X=Cl or Br, Examples include: didodecyldimethylammonium bromide (DDAB), dihexadecyldimethyl ammonium chloride, dihexadecyldimethyl ammonium bromide, dioctadecyldimethyl ammonium chloride, dieicosyldimethyl ammonium chloride, didocosyldimethyl ammonium chloride, dicoconutdimethyl ammonium chloride, ditallowdimethyl ammonium bromide (DTAB). Commercially available examples include, but are not limited to: ADOGEN, ARQUAD, TOMAH, VARIQUAT. See also US 6013683 Hill et al., .

Suitable silicone surfactants include, but are not limited to the polyalkyleneoxide polysiloxanes having a dimethyl polysiloxane hydrophobic moiety and one or more hydrophilic polyalkylene side chains and have the general formula:



wherein a + b are from about 1 to about 50, preferably from about 3 to about 30 , more preferably from about 10 to about 25, and each R<sup>1</sup> is the same or different and is selected from the group consisting of methyl and a poly(ethyleneoxide/propyleneoxide) copolymer group having the general formula:



with at least one R<sup>1</sup> being a poly(ethyleneoxide/propyleneoxide) copolymer group, and wherein n is 3 or 4, preferably 3; total c (for all polyalkyleneoxy side groups) has a value of from 1 to about 100, preferably from about 6 to about 100; total d is from 0 to about 14, preferably from 0 to about 3; and more preferably d is 0; total c+d has a value of from about 5 to about 150, preferably from about 9 to about 100 and each R<sup>2</sup> is the same or different and is selected from the group consisting of hydrogen, an alkyl having 1 to 4 carbon atoms, and an acetyl group, preferably hydrogen and methyl group. Examples of

these surfactants may be found in US 5705562 Hill and US 5707613 Hill, both of which are incorporated herein by reference.

Examples of this type of surfactants are the Silwet<sup>®</sup> surfactants which are available CK Witco, OSi Division, Danbury, Connecticut. Representative Silwet surfactants are as follows.

Name	Average MW	Average a+b	Average total c
L-7608	600	1	9
L-7607	1,000	2	17
L-77	600	1	9
L-7605	6,000	20	99
L-7604	4,000	21	53
L-7600	4,000	11	68
L-7657	5,000	20	76
L-7602	3,000	20	29

The molecular weight of the polyalkyleneoxy group ( $R^1$ ) is less than or equal to about 10,000. Preferably, the molecular weight of the polyalkyleneoxy group is less than or equal to about 8,000, and most preferably ranges from about 300 to about 5,000. Thus, the values of c and d can be those numbers which provide molecular weights within these ranges. However, the number of ethyleneoxy units ( $-C_2H_4O$ ) in the polyether chain ( $R^1$ ) must be sufficient to render the polyalkyleneoxide polysiloxane water dispersible or water soluble. If propyleneoxy groups are present in the polyalkyleneoxy chain, they can be distributed randomly in the chain or exist as blocks. Preferred Silwet surfactants are L-7600, L-7602, L-7604, L-7605, L-7657, and mixtures thereof. Besides surface activity, polyalkyleneoxide polysiloxane surfactants can also provide other benefits, such as antistatic benefits, and softness to fabrics.

The preparation of polyalkyleneoxide polysiloxanes is well known in the art. Polyalkyleneoxide polysiloxanes of the present invention can be prepared according to the procedure set forth in U.S. Pat. No. 3,299,112, incorporated herein by reference.

Another suitable silicone surfactant is SF-1488, which is available from GE silicone fluids.

These and other surfactants suitable for use in combination with the lipophilic fluid as adjuncts are well known in the art, being described in more detail in Kirk Othmer's Encyclopedia of Chemical Technology, 3rd Ed., Vol. 22, pp. 360-379, "Surfactants and Detergent Systems", incorporated by reference herein. Further suitable nonionic detergent surfactants are generally disclosed in U.S. Patent 3,929,678, Laughlin et al., issued December 30, 1975, at column 13, line 14 through column 16, line 6, incorporated herein by reference.

The adjunct may also be an antistatic agent. Any suitable well-known antistatic agents used in laundering and dry cleaning art are suitable for use in the methods and compositions of the present invention. Especially suitable as antistatic agents are the subset of fabric softeners which are known to provide antistatic benefits. For example those fabric softeners which have a fatty acyl group which has an iodine value of above 20, such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium methylsulfate. However, it is to be understood that the term antistatic agent is not to be limited to just this subset of fabric softeners and includes all antistatic agents.



What is claimed is:

1. An automatic lipophilic fluid laundry process, said process characterized by contacting a fabric in an automatic lipophilic fluid laundry medium with a lipophilic fluid-containing wash medium and one or more coloring agents, wherein said coloring agents are safe for use in said process.
2. The process according to Claim 1 wherein the coloring agent is dissolved or dispersed in the lipophilic fluid or in the siloxane solvent.
3. The process according to any of the preceding claims wherein the lipophilic fluid is a mixture of siloxane solvent and water, preferably wherein the mixture, when present in the wash medium further comprises an emulsifier and is in the form of an emulsion.
4. The process according to any of the preceding claims further characterized by the process of removing some or all of the coloring agent from the lipophilic fluid-containing wash medium, preferably whereby at least part of the coloring agent is removed by filtration, distillation, or combinations thereof or at least part of the coloring agent is destroyed as part of the removal process, preferably by chemical means, mechanical means, electrical means, thermal means, or combinations thereof.
5. The process according to any of the preceding claims wherein at least a part of the coloring agent is stable to the laundry process and lipophilic fluid recovery process.
6. A composition for use in an automatic home laundry process, said composition characterized by a coloring agent safe for use in a siloxane solvent-containing wash medium and an adjunct ingredient selected from emulsifiers for a siloxane-containing wash medium, surfactants for a siloxane-containing wash medium, and mixtures thereof.

7. An automatic lipophilic fluid laundry process, said process characterized by contacting a fabric in an automatic lipophilic fluid laundry medium with a lipophilic fluid-containing wash medium and one or more sudsing agents.
8. The process according to Claim 7 wherein the lipophilic fluid-containing wash medium is characterized by siloxane solvent.
9. The process according to Claims 7-8 further characterized by the process of removing some or all of the sudsing agent from the lipophilic fluid-containing wash medium.
10. The process according to Claims 7-9 whereby at least part of the sudsing agent is removed by filtration, distillation, or combinations thereof or at least part of the sudsing agent is destroyed as part of the removal process, preferably by chemical means, mechanical means, electrical means, thermal means, or combinations thereof.
11. The process according to Claims 7-10 wherein the lipophilic fluid-containing wash medium is characterized by siloxane solvent and at least 0.1% water.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 01/18192

A. CLASSIFICATION OF SUBJECT MATTER		
IPC 7	D06L1/04	C11D7/50 C11D3/43 C11D3/40 C11D3/16
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols)		
IPC 7 D06L C11D		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the International search (name of data base and, where practical, search terms used)		
WPI Data, EPO-Internal, PAJ		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 091 261 A (HOLLYGRANGE LTD) 12 October 1983 (1983-10-12) abstract	1,2,7
A	page 4, line 6 - line 20; examples	6
X	US 6 063 135 A (BERNDT DIETER R ET AL) 16 May 2000 (2000-05-16) cited in the application	7-10
A	claims 1,2; figure 1	1-4,6,11
X	DE 37 39 711 A (KREUSSLER CHEM FAB) 8 June 1989 (1989-06-08)	7-11
A	page 2, line 58 - line 61; example 2	1-3
X	WO 82 02218 A (ELECTROLUX AB) 8 July 1982 (1982-07-08)	7,9,10
A	claims 1,8,9; examples	1,3
-/-		
<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents : *A* document defining the general state of the art which is not considered to be of particular relevance *E* earlier document but published on or after the international filing date *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) *O* document referring to an oral disclosure, use, exhibition or other means *P* document published prior to the international filing date but later than the priority date claimed *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art *Z* document member of the same patent family		
Date of the actual completion of the international search		Date of mailing of the international search report
18 October 2001		26/10/2001
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer  Saunders, T

# INTERNATIONAL SEARCH REPORT

Interp - 121 Application No  
PC1/US 01/18192

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Section Ch, Week 199704 Derwent Publications Ltd., London, GB; Class A97, AN 1997-038179 XP002180007	7
A	-& JP 08 295897 A (KAO CORP), 12 November 1996 (1996-11-12) abstract	1
X	US 3 635 667 A (KEAY ROBERT E ET AL) 18 January 1972 (1972-01-18)	7
A	claim 1	1
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A	abstract; example 1	1,5,7

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